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(54) PRODUCTION OF SINTERED METALLIC PART BY INJECTION MOLDING OF METAL POWDER

(57)Abstract:

PURPOSE: To produce a sintered metallic part increased in strength, hardness, etc., and excellent in dimensional accuracy by mixing a resin in a metal powder as a binder, compacting the mixture, degreasing the compact under specified conditions and sintering the compact.

CONSTITUTION: Thermoplastic resin such as PE as a binder, wax as a low-mol. wt. org. matter, etc., are added to the powder of steel copper and other metals and alloys having $\leq 100\mu\text{m}$ grain diameter and mixed to produce an injection-molding green compound. The compound is molded into a desired shape by an injection-molding machine, and the compact is heated to about 300°C in a closed furnace contg. an inert gas or hydrogen atmosphere to volatilize off the low-mol.wt. component such as wax, and then heated to $600-1000^\circ\text{C}$ to decompose the resin into hydrocarbons. The compact is carburized by the hydrocarbons to remove oxides and then sintered at $800-2000^\circ\text{C}$, and a sintered metallic part increased in strength and hardness and excellent in dimensional accuracy is produced.

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(54)【発明の名称】 金属粉末射出成形法による焼結金属部品の製造方法

(57)【要約】

【目的】脱脂工程の一部を600℃～1000℃の温度で、密閉した加熱炉内で行うことにより、脱脂後のC量を適切に調整し、焼結体の寸法精度を向上させた金属粉末射出成形法による焼結金属部品の製造方法の提供。

【構成】金属粉末射出成形法により金属焼結部品を製造する工程において、脱脂工程の少なくとも一部を600℃以上、1000℃以下の温度で行い、かつ、その時の熱処理を密閉した加熱炉内で行う焼結金属部品の製造方法。

【特許請求の範囲】

【請求項1】金属粉末射出成形法により金属焼結部品を製造する工程において、脱脂工程の少なくとも一部を600℃以上、1000℃以下の温度で行い、かつ、その時の熱処理を密閉した加熱炉内で行うことを特徴とする焼結金属部品の製造方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、金属粉末射出成形法による焼結金属部品の製造方法に関する。

【0002】

【従来の技術】金属粉末射出成形法は、小型複雑形状の金属部品を大量生産する方法として利用される技術である。この方法においては、まず原料である金属粉末とバインダを混練して、射出成形用原料コンパウンドとする。このコンパウンドは熱可塑性を有し、射出成形機によって所望の形状に成形される。この成形工程は、本質的にプラスチック材料の成形と変わる所がなく、大量成形が可能である。次に、得られた成形体から不要のバインダを除去する。この工程を脱脂という。脱脂方法には、加熱してバインダを成形体から蒸発、あるいは流出させる方法、溶媒中に成形体を保持してバインダを抽出する方法、またこの二つを組み合わせる方法などが知られており、バインダの種類に応じて脱脂方法は選ばれる。最後に脱脂体を焼結して金属部品を得る。

【0003】金属粉末射出成形法は、高い焼結密度が得られる金属微粉を成形できることに特徴がある。従来、平均粒径が10μm以下であるような微粉は、流動性が悪いという問題や、金型のかじりの問題からプレスによる成形が困難であったが、この方法によれば微粉でも容易に成形ができ、しかも3次元的な複雑形状まで成形可能である。このような利点を持つことから、最近ステンレス部品の製造などにも金属粉末射出成形法が利用されることが多くなり、磁性材料や超硬材料にも応用が広がっている。

【0004】ところで、焼結金属部品の特性は、その含有炭素(C)量に大きく影響される。例えば、Fe-Ni系などの機械構造用部品の強度や硬さはC量に大きく支配され、ステンレス鋼においては、機械的性質はむしろ耐食性もC量に影響される。同様なことが焼結体の酸素(O)量についても言える。したがって、焼結部品の製造においてもC、O量を所望の量に制御する技術が要求される。金属粉末射出成形法においては、最終的な焼結体のC、O量は粉末のC、O量、バインダの種類、脱脂方法、焼結方法、部品の形状に影響される。C、O量の調整方法としては、粉末のC、O量によって調整する方法はもちろん、O量の調整法として、酸化物を添加する方法(特開平2-57607号公報)が開示されている。C量については、C量を低減する場合に

する方法(特開平3-45566号公報)、Cを積極的に含有させたい場合には、原料粉末にグラファイトを添加する方法(特開平2-39402号公報)などが開示されている。

【0005】

【発明が解決しようとする課題】しかし、グラファイトを添加する方法は、グラファイトと金属粉末が均一に混ざりにくく偏析が起こるという問題や、グラファイト粉末がフレーク状であるため、射出成形時には原料の流動方法に配向しやすく、結果的に焼結体の不均一収縮の原因となって焼結体の寸法精度が低下してしまうという問題がある。

【0006】

【課題を解決するための手段】以上の問題を鑑みて、本発明は、金属粉末射出成形法で製造される焼結体のC量を適切に調整し、寸法精度を向上することのできる焼結金属部品の製造方法を見だし、本発明に至った。

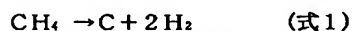
【0007】すなわち、本発明は、焼結体のC量を調整するために、脱脂工程の一部を600℃以上、1000℃以下の温度で行い、かつ、その時の熱処理を密閉した加熱炉内で行うことを特徴とする焼結金属部品の製造方法を提供する。

【0008】本発明によって、脱脂工程で所望のC量を添加することができ焼結体のC量が制御可能となる。

【0009】

【作用】以下、本発明をさらに詳しく説明する。金属粉末射出成形法では、原料金属粉末を有機バインダと混練してなる原料コンパウンドを射出成形法によって部品形状に成形する。バインダは、成形のための助剤であるから、脱脂工程で取り除く必要がある。バインダには通常、熱可塑性樹脂が含まれるが、この樹脂は300~500℃で熱分解によって除去される。本発明の特徴は、この樹脂分解時の脱脂工程の熱処理を、600℃以上1000℃以下の温度範囲で、かつ、その時の熱処理を密閉した加熱炉内で行うことにある。

【0010】熱可塑性樹脂などの高分子化合物は、熱によって分解して低分子のガス(分解ガス)となる。その分解挙動は、樹脂の種類によって様々であるが、分解して生じた分解ガスは、本質的にはC₁~C₆の炭化水素であると考えられる。このような炭化水素は、一般に高温下では金属に対して浸炭作用をもつ。例えば、メタン(CH₄)においては、



の反応によって金属に浸炭するか、あるいは金属表面にフリーのカーボンとしてCを存在させる。この反応挙動は、炭素数の多い炭化水素についても同様である。したがって、脱脂時に、浸炭反応が起こるに十分な温度に昇温してやれば、脱脂体にCを残すことが可能である。一方、従来の射出成形体の脱脂工程では、樹脂が分解するのに必要な温度域で脱脂を行い、分解ガスをすみやかに

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除去するように窒素等の不活性ガスを流すのが普通であった。しかし、本発明では、分解ガスを積極的にC源として利用することを目的としているので、脱脂時には炉を密閉することが肝要である。

【0011】本発明によって、処理後の脱脂体のC量を原料粉末よりも多くすることができるが、その増加量は、熱処理時の加熱温度と処理時間によって制御可能である。すなわち、加熱温度が高いほど、また、処理時間が長いほど、脱脂体のC量は多くなる。C量の増加量の絶対値は、粉末の鋼種や粉末特性およびバインダの種類に依存するが、温度と処理時間によって制御できることには変わりがない。

【0012】さらに、本発明によれば、脱脂体に均一にCを分布させることができる。したがって、グラファイトを添加する方法の問題点であった、Cの偏析や、前述した不均一収縮も起こらない。

【0013】以上に述べたように、脱脂工程の一部を600℃以上1000℃以下の温度で行い、かつ、その時の熱処理を密閉した加熱炉内で行うことによって、脱脂体に所望のC量を均一に増加させることができ、その上、歪のない寸法精度に優れた焼結体を製造することができる。

【0014】本発明に用いる射出成形体は、金属粉末と有機バインダを含有する。

【0015】本発明に利用できる金属粉末は、鉄、銅、チタン、タングステン、ニッケル、モリブデン、クロムの粉末、あるいはステンレス鋼、Fe-Ni系合金などの合金粉末が挙げられる。また、粉末の製造法別に挙げると、カルボニル粉、水アトマイズ粉、ガスアトマイズ粉、粉砕粉などを挙げるることができる。さらに、これらのうちの2種以上の混合粉を用いることも可能である。粉末の粒度は0.01~1000μmの範囲が使用可能であるが、コンパウンドの流動性、あるいは粉末の焼結性から100μm以下の粉末を使用するのが好ましい。さらに好ましい粒径の範囲は0.1~50μmである。

【0016】本発明に利用できる有機バインダの成分としては、熱可塑性樹脂、ワックス、可塑剤、滑剤などが挙げられる。樹脂は、バインダの主成分として原料コンパウンドに可塑性を与え、また常温での成形体強度を持たせる。樹脂成分としては、ポリエチレン、ポリプロピレン、ポリスチレン、エチレン-酢酸ビニル共重合体(EVA)、エチレン-エチルアクリレート共重合体(EEA)、ポリメタクリル酸アルキルエステル、ポリアミドなどが公知であって、これらのうち1種または2種以上を組み合わせて用いられる。

【0017】さらに、脱脂性、流動性の改善のために、樹脂よりも低分子の有機物であるワックスや可塑剤などを添加する。樹脂よりも低分子の成分としては、パラフィンワックス、高級脂肪酸、高級アルコール、高級脂肪酸エステル、高級脂肪酸アミド、フタル酸ジエチル、フ

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タル酸ジブチル等のフタル酸エステルなどの1種または2種以上を組み合わせ用いられる。このように金属粉末射出成形用原料コンパウンドは、金属粉末と樹脂、ワックス、可塑剤など数種の有機物の混合物であるのが一般的である。

【0018】有機バインダと金属粉末との配合比は、金属粉末100重量部に対して、有機バインダ3~20重量部が好ましい。3重量部未満ではバインダが金属粉末に対して不足し、コンパウンドの流動性が悪く射出成形が困難となる。また、20重量部を超えると、射出成形はできるものの、脱脂時の形状保持ができなくなるので好ましくない。

【0019】これらの金属粉末と有機バインダを混練して、射出成形用コンパウンドを製造する。混練機としては、ヘンシェルミキサー、プラストミル、加圧ニーダー、パンバリーミキサー、ロールミル、単軸スクリュ混練機、2軸スクリュ混練機などが使用でき、これらの2種以上を組み合わせ混練してもよい。

【0020】混練したコンパウンドは、粉砕あるいは造粒して成形材料とする。射出成形機は、一般的な熱可塑性プラスチック用射出成形機を用いることができる。射出成形は、射出温度100℃~250℃の範囲で行うが、射出温度が高すぎるとバインダ成分の変質が顕著になり、再生材の成形性、脱脂性の変化をきたすので、好ましくは射出温度は100℃~180℃の範囲がよい。脱脂は、脱脂炉中、加熱によって行う。

【0021】本発明で行なう脱脂の工程は、樹脂以外の低分子成分を除去する前処理の工程と、成形体のC量を調節する工程とからなる。

【0022】前処理の工程として、欠陥や形状変形の無い脱脂体を得るためには、樹脂以外の低分子成分を樹脂分解温度以下で徐々に除去することが必要である。これは大気、窒素やアルゴンなどの不活性ガス、水素などの還元性ガスでの雰囲気下で、昇温速度10℃/h~100℃/hの範囲で昇温することによって行われる。また、雰囲気として真空も利用できる。この前処理は、300℃以下で行うのが好ましい。また、バインダ成分の一部を溶媒で抽出する方法を用いてもよい。

【0023】本発明においては、成形体のC量を調節するために、前処理後の脱脂工程の一部を600℃以上1000℃以下で行い、さらに、その時には密閉した加熱炉内で行う。

【0024】具体的には、低分子成分を除去した後に、還元性または不活性ガスを導入していた場合には、ガスの導入を停止した後、排気のための導管のバルブも閉じ加熱炉内を密閉する。好ましくは、還元性または不活性ガスの導入を停止した後は、加熱炉内の圧力を10 Torr以下にしてから加熱炉内を密閉するのがよい。これは、その後の昇温のときの圧力の上昇を抑えるためである。加熱炉を密閉した後、昇温を行い、樹脂を分解させ

る。生じた分解ガスによって脱脂体のC量を増加させる。

【0025】また、低分子成分を除去した後に、600～1000℃に昇温後、同様の方法で加熱炉内を密閉しても本発明と同じ効果が得られる。

【0026】処理温度と処理時間は、金属粉末の組成や粉末特性、バインダ種および増加させたいC量によって選択する。しかし、処理温度は、600～1000℃の範囲であるのが好ましい。処理温度が600℃未満では、効率的にC量の増加が起こらず、また、1000℃を超えると、粉末の焼結が進行し脱脂体内に均一にガスがいきわたらずC量の不均一分布が発生するので好ましくない。さらに、焼結が進行すると分解ガスが脱脂体内にトラップされ焼結体の特性に悪影響を及ぼすこともある。したがって、処理温度は600℃以上、1000℃以下、好ましくは700～900℃の範囲で行うのがよい。

【0027】焼結工程は、脱脂工程終了後、引き続き同一炉内で行ってもよいし、脱脂体を脱脂炉から取り出し後、異なる炉で行ってもよい。同一炉内で行うときは、炉内を排気してから昇温を行うのが好ましい。焼結は、800℃～2000℃の温度で、10分～6時間保持して行うが、これら焼結条件、焼結雰囲気は用いる金属粉末の材質、粉末特性に応じて適宜選択して決める。

【0028】

【実施例】

(実施例1) 平均粒径8.5μm、C含有量：0.03wt%、O含有量：0.70wt%のSUS316組成の水アトマイズ粉を用意した。これに、ポリエチレン40wt%、ポリブチルメタクリレート10wt%、パラフィンワックス30wt%、ジブチルフタレート20wt%からなるバインダを、金属粉末100重量部に対し10重量部添加し、加圧ニーダを用いて混練して射出成形用コンパウンドを製造した。このコンパウンドを射出成形機によって、4mm×10mm×50mmの直方体の試験片に成形した。

【0029】この試験片(成形体)を、下記のとおり脱脂焼結した。脱脂の前処理の工程として、窒素気流中、常温から250℃まで40時間で昇温し、パラフィンワックスとジブチルフタレートの大部分を除去した。その後、窒素の導入を停止し、さらに排気側のバルブを閉じて、別系統の配管によってロータリーポンプで炉内を1 Torrまで減圧し、炉を密閉した。その後、脱脂処理の最高温度を550℃～1050℃まで変化させて脱脂体を作製し、発明例1～6の脱脂体を得た。具体的には、最高温度まで1時間で昇温し、各発明例の最高温度で1時間保持後、炉内を排気するとともに冷却して脱脂体を得た。最高温度を550℃～1050℃と変化させたときの脱脂体のC量を分析した結果を表1に示す。加熱温度が高くなるほど有機バインダから発生した分解ガ

スの浸炭作用によって脱脂体のC量が増加している。焼結時にCによる還元反応によって、粉末の酸素を除くにはC量として0.30～0.45%の範囲が適切であり、焼結体も好ましい特性(C：0～0.05%、O：0～0.30%)が得られる。ここでは、最高加熱温度850℃の脱脂条件のもの(発明例4)を実施例1として、焼結体の評価を行った。

【0030】焼結は、真空下で、1320℃まで10℃/分で昇温し、2時間保持後冷却して行った。この焼結体を、実施例1の焼結体とする。焼結体の評価として、幅方向と長さ方向の寸法を測定するとともに、C、Oの化学分析を行った。結果を表2に付記する。脱脂の最高温度が1000℃を超える脱脂体を焼結したところ、焼結体のC量が高くなり好ましい焼結体を得られなかった。また、脱脂温度が600℃未満では、焼結体のO量が低減されておらず、やはり好ましい焼結体を得られな

【0031】(比較例1) 実施例1で使用したSUS316組成の水アトマイズ粉に、グラファイト粉末0.35wt%を混合した粉末を原料粉末として用いる以外は、実施例1と同様に混練、成形を行った。脱脂は、窒素気流中、常温から250℃まで40時間で昇温した後に、1時間かけて500℃まで昇温し、500℃で1時間保持した。脱脂工程は、すべて窒素を流しながら窒素気流中で行った。焼結は、実施例1と同様に行い、評価した。

【0032】(比較例2) 比較例1で使用したグラファイトを混合したSUS316組成の水アトマイズ粉を用いて、実施例1と同じ工程で焼結体を製造して、その評価を行った。

【0033】(比較例3) 脱脂をすべて窒素を流しながら窒素気流中で行う以外は、実施例1と全く同じ射出成形用コンパウンドを用い、同じ工程で焼結体を製造して、その評価を行った。

【0034】実施例1と比較例1、2での焼結体の評価結果を表2に示す。

【0035】実施例1では、本発明による脱脂処理を行っているので、脱脂後にCが増加しており、焼結体の特性は好ましいものであった。比較例1では、同じ目的で原料粉末にグラファイト粉末を添加して焼結体を製造したものである。特性的には実施例1と変わらず好ましいものであるが、長さ方向と幅方向の収縮率の差は2.5%と実施例1の0.5%にくらべて大きく、寸法精度に劣っていた。比較例2では、脱脂体のC量が過剰であり、特性の好ましい焼結体を得られなかった。さらに長さ方向と幅方向の収縮率の差は3.2%と寸法精度が良くなかった。比較例3は、実施例1の脱脂工程をすべて窒素気流中で行ったので脱脂工程でのCの増加が少なく、焼結体のO量が多くなり好ましい焼結体を得られなかった。

【0036】

* * 【表1】

表 1

	発明例1	発明例2	発明例3	発明例4 (実施例1)	発明例5	発明例6
脱脂加熱最高温度 (°C)	550	600	750	850	1000	1050
脱脂体C量 (重量%)	0.15	0.30	0.33	0.40	0.45	0.95
焼結体C量 (重量%)	0.01	0.01	0.02	0.03	0.05	0.15
焼結体O量 (重量%)	0.55	0.30	0.25	0.23	0.18	0.08

【0037】

※ ※ 【表2】

表 2

	焼結体密度 (%)	脱脂体 C量 (重量%)	焼結体 C量 (重量%)	焼結体 O量 (重量%)	長さ方向と幅方向の 収縮率の差 (%)
実施例1	97.7	0.40	0.03	0.23	0.5
比較例1	97.5	0.38	0.02	0.24	2.5
比較例2	95.2	0.85	0.17	0.10	3.2
比較例3	96.4	0.10	0.01	0.55	0.7

【0038】 (実施例2) カルボニル鉄粉とカルボニルニッケル粉を原料として、Fe-4%Ni組成の混合粉末を用意した。混合粉末の平均粒径は、5.5 μ m、C含有量:0.05wt%、O含有量:0.25wt%であった。この混合粉末に実施例1と同じ配合の有機バインダを9重量部添加して、実施例1と同様に混練、成形、脱脂、焼結を行い、焼結体を評価した。

【0039】 (比較例4) 実施例2のFe-4%Ni組成の混合粉末に、グラファイト粉末を0.5wt%添加する以外は、実施例2と同様にして混練、成形を行っ
30 た。脱脂は、窒素気流中、常温から250°Cまで40時間で昇温した後、1時間で500°Cまで昇温し、500★

★℃で1時間保持した。脱脂工程は、すべて窒素を流しながら窒素気流中で行った。焼結は、実施例1と同様に行い、脱脂体および焼結体を評価した。

【0040】 実施例2と比較例3での焼結体の評価結果を表3に示す。

【0041】 本発明による方法でもグラファイトを添加する方法でも高密度でC量を制御した焼結体を得ることができた。しかし、グラファイトを添加する方法では長さ方向と幅方向の収縮率の差が大きく、本発明による焼結体のほうが、寸法精度に優れていることがわかる。

【0042】

【表3】

表 3

	焼結体密度 (%)	脱脂体 C量 (重量%)	焼結体 C量 (重量%)	焼結体 O量 (重量%)	長さ方向と幅方向の 収縮率の差 (%)
実施例2	97.7	0.67	0.52	0.002	0.3
比較例4	97.4	0.65	0.51	0.002	2.1

【0043】

【発明の効果】 本発明の製造方法によれば、金属粉末射出成形法の脱脂工程の一部を600°C以上、1000°C
40

以下の温度で行い、かつ、その時の熱処理を密閉した加熱炉内で行うことによって、脱脂後のC量を適切に調整することを可能にし、焼結体の寸法精度も向上した。

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(54) [Title of the invention] Method for producing sintered metal parts by injection moulding metal powder

(57) [Abstract]

[Aim] The provision of a method for producing sintered metal parts by injection moulding metal powder such that the sintered body has improved dimensional accuracy, where the C content after degreasing is adjusted as appropriate by performing some of the degreasing process at a temperature of from 600°C to 1000°C in a closed heated furnace.

[Constitution] A method for producing sintered metal parts, where in a process for producing metal sintered parts by injection moulding metal powder, at least some of the degreasing process is performed at a temperature of from 600°C to 1000°C, and at that time the heat treatment is performed in a closed heated furnace.

[Scope of the patent claim]

[Claim 1] A method for producing sintered metal parts, characterised in that in a process for producing metal sintered parts by injection moulding metal powder, at least some of the degreasing process is performed at a temperature of from 600°C to 1000°C, and at that time the heat treatment is performed in a closed heated furnace.

[Detailed description of the invention]

[0001]

[Field of industrial use] The present invention relates to a method for producing sintered metal parts by injection moulding metal powder.

[0002]

[Prior art] The injection moulding of metal powder is used as a method for mass-producing small, complex-shaped metal parts. In this method, first the starting materials - metal powder and binder - are mixed to form a starting material compound for injection moulding. This compound is thermoplastic, and can be formed into the desired shape using an injection moulder. This moulding process does not differ fundamentally from the moulding of plastic materials, and mass-moulding is possible. Next, unnecessary binder is removed from the resulting moulding. This process is referred to as degreasing. Known degreasing methods include methods whereby binder is vaporised off from or run-off from the moulding by heating, methods whereby the moulding is kept in a solvent such that the binder is extracted, and combinations of these two methods; the degreasing method is chosen according to the type of binder. Finally, the degreased body is sintered to yield a metal part.

[0003] A special feature of the metal powder injection moulding method is that it allows the moulding of fine metal powders that result in a high sintering density. Conventionally, fine powder having an average particle diameter no greater than 10 μm is disadvantageous in that its fluidity is poor, and because of problems with galling of the mould, it is difficult to mould by pressing; with this method, however, it is easy to mould even fine powder, and moulding into 3-dimensional complex shapes is also possible. In view of these advantages, the metal powder injection moulding method has recently become more frequently employed, even in the production of stainless steel parts, and so forth, and it is also becoming widely used for magnetic materials and extra hard materials.

[0004] The properties of sintered metal parts are greatly affected by the amount of carbon (C) they contain. For example, the strength and hardness of parts for mechanical construction comprising Fe-Ni and the like are governed largely by the C content, and the C content of stainless steel affects its mechanical properties of course, but also its corrosion resistance. The same can also be said of the amount of oxygen (O) in the sintered body. Consequently there is demand for a technique for controlling the amounts of C and O as desired in sintered part production. With the metal powder injection moulding method, the amounts of C and O in the final sintered body depend on the amounts of C and O in the powder, the type of binder, the degreasing method, the sintering method and the shape of the part. Adjusting the amounts of C and O in the powder has of course been disclosed as a method for adjusting the C and O contents, and adding oxide (Japanese unexamined patent H2-57607) has been disclosed as a method for adjusting the O

content. As for the C content, removing carbon by introducing hydrogen gas during degreasing (Japanese unexamined patent H3-45566) has been disclosed as a method for lowering the C content, and adding graphite to the starting material powder (Japanese unexamined patent H2-39402) has been disclosed as a method for when C is to be positively introduced, for example.

[0005]

[Problems to be resolved by the invention] However, adding graphite is disadvantageous in that it is difficult to mix graphite and metal powder uniformly and segregation occurs, and because graphite powder is flaky, it readily becomes oriented in the method [sic] of the flow of the starting material during injection moulding, which results in uneven shrinkage of the sintered body such that the dimensional accuracy of the sintered body is decreased.

[0006]

[Means of resolving the problems] In view of the above-mentioned problems, the present invention was realised by the discovery of a method for producing sintered metal parts, whereby dimensional accuracy can be improved by appropriately adjusting the C content of a sintered body produced by injection moulding metal powder.

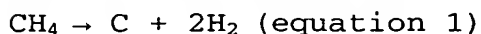
[0007] Specifically, the present invention provides a method for producing sintered metal parts, characterised in that in order to adjust the C content of the sintered body, some of the degreasing process is performed at a temperature of from 600°C to 1000°C, and at that time the heat treatment is performed in a closed heated furnace.

[0008] With the present invention, the desired amount of C can be added during the degreasing process and the C content of the sintered body can be controlled.

[0009]

[Action] The present invention is described in more detail below. In the metal powder injection moulding method, starting material metal powder is mixed with organic binder, and the resulting starting material compound is moulded into the shape of the part by the injection moulding method. The binder is an auxiliary agent for moulding and must therefore be removed by the degreasing process. The binder usually contains thermoplastic resin, and the resin is removed by thermal decomposition at from 300 to 500°C. The special feature of the present invention is that the degreasing process heat treatment during the resin decomposition is performed at a temperature of from 600°C to 1000°C, and at that time the heat treatment is performed in a closed heated furnace.

[0010] High molecular weight compounds such as thermoplastic resins decompose on heating to form low molecular weight gas (decomposition gas). There are various decomposition mechanisms depending on the type of resin, and the decomposition gas produced on decomposition is thought to be essentially C₁ - C₆ hydrocarbons. These hydrocarbons usually have a carburising effect on metal at high temperatures. With methane (CH₄), for example, metal is carburised according to the reaction



or C is present as free carbon on the surface of the metal. The reaction mechanism is similar for hydrocarbons containing more carbon atoms. Accordingly, if the temperature during degreasing is high enough for carburisation to occur, C may remain in the degreased body. Furthermore, in the conventional injection moulding degreasing process, degreasing is performed in the

temperature range necessary for resin decomposition, and it is usually performed in a flow of inert gas such as nitrogen, to quickly remove the decomposition gas. However, in the present invention, the aim is to use the decomposition gas positively as a source of carbon, and so it is important that the furnace is closed during degreasing.

[0011] With the present invention, it is possible to increase the C content of the post-treatment degreased body so that it is greater than that of the starting material powder, and this increase in C content can be controlled by means of the heating temperature and treatment time employed during the heat treatment. Specifically, the higher the heating temperature, and the longer the treatment time, the greater the C content in the degreased body. The absolute value for the increase in C content depends on the powder steel type, the properties of the powder and the type of binder, but can always be controlled by means of temperature and treatment time.

[0012] Furthermore, C can be distributed uniformly in the degreased body by means of the present invention. Accordingly, there is neither C segregation, which is a problem with the method whereby graphite is added, nor the above-mentioned non-uniform shrinkage.

[0013] As described above, by performing some of the degreasing process at a temperature of from 600°C to 1000°C, and performing the heat treatment at that time in a closed heated furnace, the degreased body C content can be uniformly increased as desired, and in addition, a sintered body of excellent dimensional accuracy with no distortion can be produced.

[0014] The injection moulding used in the present invention contains metal powder and organic binder.

[0015] Metal powders that can be used in the present invention are powders of iron, copper, titanium, tungsten, nickel, molybdenum and chromium, and powders of alloys such as stainless steel and Fe-Ni alloy. Or, to classify according to the method for producing the powder, they can be carbonyl powders, water-atomised powders, gas-atomised powders, pulverised powders and the like. Moreover, mixtures comprising 2 or more of these can be used. The powder particle size can be in the range of from 0.01 to 1000 μm , although for compound fluidity and the sintering properties of the powder, it is preferable to use a powder having a particle size no greater than 100 μm . The particle size is more preferably from 0.1 to 50 μm .

[0016] Examples of organic binder components that can be used in the present invention include thermoplastic resins, waxes, plasticisers and lubricants. The resin, as the main component of the binder, bestows elasticity to the starting material compound and affords moulding strength at room temperature. Known resin components include polyethylene, polypropylene, polystyrene, ethylene-vinyl acetate copolymer (EVA), ethylene-ethyl acrylate copolymer (EEA), polymethacrylate alkyl ester and polyamide, and 1 of these, or combinations of 2 or more of these, can be used.

[0017] An organic substance having a lower molecular weight than the resin, such as wax, plasticiser or the like, is added to improve the degreasing properties and fluidity. Paraffin wax, higher fatty acids, higher alcohols, higher fatty acid esters, higher fatty acid amides and phthalate esters such as diethyl phthalate and dibutyl phthalate are used as the component having a lower molecular weight than

the resin, and 1 of these, or combinations of 2 or more of these, can be used. The starting material compound for metal powder injection moulding is usually a mixture of metal powder and a plurality of organic substances such as resin, wax and plasticiser.

[0018] The proportion of organic binder to metal powder is preferably from 3 to 20 parts by weight of organic binder per 100 parts by weight of metal powder. Less than 3 parts by weight of binder is insufficient with respect to the metal powder, such that the fluidity of the compound is poor and injection moulding becomes difficult. If there are more than 20 parts by weight, injection moulding is possible but shape retention during degreasing becomes impossible, and so the use of such amounts is undesirable.

[0019] The compound for injection moulding is produced by mixing the metal powder and the organic binder. Mixers that can be used include the Henschel mixer, plast mill, pressure kneader, Banbury mixer, roll mill, single-screw mixer and twin-screw mixer, and the mixing may involve the use of a combination of 2 or more of these.

[0020] The mixed compound is pulverised or granulated to form the moulding material. A common injection moulder for thermoplastic plastics can be used as the injection moulder. The injection moulding is performed at an injection temperature of from 100°C to 250°C; however, if the injection temperature is too high, there is marked deterioration of the binder component, which alters the moulding properties and degreasing properties of the regenerated material, and so the injection temperature is preferably from 100°C to 180°C. The degreasing is performed by heating in a degreasing furnace.

[0021] The degreasing process in the present invention comprises a pre-treatment process whereby low molecular weight components other than the resin are removed, and a process whereby the C content of the moulding is regulated.

[0022] In the pre-treatment process, low molecular weight components other than the resin must be gradually removed at below the resin decomposition temperature, in order to obtain a degreased body with no defects or shape deformation. This is achieved by heating at a rate of from 10°C/hour to 100°C/hour in an atmosphere of an inert gas such as air, nitrogen or argon, or of a reducing gas such as hydrogen. A vacuum may also be employed as the atmosphere. This pre-treatment preferably proceeds at no greater than 300°C. Furthermore, an extraction method may be employed, using some of the binder component as solvent.

[0023] In order to regulate the C content of the moulding in the present invention, the part of the degreasing process that follows the pre-treatment is performed at from 600°C to 1000°C, and at this time the degreasing proceeds in a closed heated furnace.

[0024] Specifically, after the low molecular weight components have been removed, a reducing or inert gas is introduced, then gas introduction is stopped, after which the valve of the exhaust gas inlet pipe is closed and the inside of the heated furnace is closed. Preferably, after the introduction of reducing or inert gas has stopped, the inside of the heated furnace is closed when the pressure inside the heated furnace is no greater than 10 Torr. This is to suppress the pressure increase that occurs when the temperature is subsequently increased. After the heated furnace has been closed, the temperature is increased and

the resin is decomposed. The decomposition gas produced is used to increase the C content of the degreased body.

[0025] Also, the same advantages of the present invention are achieved even if the temperature is increased to from 600 to 1000°C after the low molecular weight components have been removed, and then the inside of the heated furnace is closed by the same method.

[0026] The treatment temperature and treatment time are selected depending on the metal powder composition and powder properties, the type of binder and the C content increase required. The treatment temperature is preferably from 600 to 1000°C. If the treatment temperature is less than 600°C, the C content is not increased efficiently, and if it exceeds 1000°C, sintering of the powder is promoted and gas does not spread uniformly in the degreased body, resulting in non-uniform C content distribution, which is undesirable. Moreover, if sintering proceeds, the decomposition gas becomes trapped in the degreased body, which adversely affects the properties of the sintered body. The treatment temperature is therefore from 600 to 1000°C, preferably from 700 to 900°C.

[0027] The sintering process may proceed after completion of the degreasing process, in the same furnace, or the degreased body may be taken from the degreasing furnace, and the subsequent sintering process may be performed in a different furnace. When the same furnace is used, it is preferable to exhaust the inside of the furnace then increase the temperature. The sintering involves keeping the system at from 800°C to 2000°C for from 10 minutes to 6 hours; the sintering conditions and sintering atmosphere are selected as appropriate depending on the metal powder material and the properties of the powder.

[0028]

[Working examples]

Working example 1

Water-atomised powder of SUS 316 composition having an average particle diameter of 8.5 μm , a C content of 0.03 wt% and an O content of 0.70 wt% was prepared. A compound for injection moulding was produced by adding 10 parts by weight of binder, comprising 40 wt% polyethylene, 10 wt% polybutyl methacrylate, 30 wt% paraffin wax and 20 wt% dibutyl phthalate, to 100 parts by weight of the metal powder, and mixing using a pressure kneader. This compound was moulded into 4 mm x 10 mm x 50 mm parallelepiped test samples using an injection moulder.

[0029] The test samples (mouldings) were degreased and sintered as follows. The degreasing pre-treatment process involved increasing the temperature from room temperature to 250°C over a period of 40 hours in a nitrogen atmosphere to remove most of the paraffin wax and dibutyl phthalate. The introduction of nitrogen was then stopped, the valve on the exhaust gas side was closed, the pressure inside the furnace was decreased to 1 Torr using a rotary pump via a separate pipeline system, and the furnace was closed. Degreased bodies for inventive examples 1 to 6 were prepared by varying the maximum degreasing treatment temperature between 550°C and 1050°C. Specifically, the temperature was increased to the maximum temperature over a period of 1 hour, the temperature was then kept at the maximum temperature for the respective inventive example for 1 hour, then the inside of the furnace was exhausted and cooled, to yield the degreased body. The C content analysis results for the degreased bodies obtained on varying the maximum temperature between 550°C and 1050°C

are shown in Table 1. The higher the temperature, the greater the degreased body C content, due to the carburising effect of the decomposition gas produced from the organic binder. From 0.30 to 0.45% is a suitable C content for removing the oxygen from the powder, during sintering, by reduction reaction due to the C present, and the resulting sintered body also has desirable properties (C: 0 to 0.05%, O: 0 to 0.30%). Here, the sample obtained by degreasing under conditions such that the maximum temperature was 850°C (inventive example 4) was used for Working example 1, and the sintered body was appraised.

[0030] Sintering was performed, in a vacuum, by increasing the temperature to 1320°C at 10°C/minute, maintaining that temperature for 2 hours, then cooling. The resulting sintered body was used as the sintered body for Working example 1. To appraise the sintered body, the length and width dimensions were measured, and C and O chemical analyses were performed. The results are shown in Table 2. When a degreased body obtained at a maximum degreasing temperature greater than 1000°C was sintered, the sintered body C content was high and the sintered body was undesirable. When the degreasing temperature was less than 600°C, there was no lowering of the sintered body O content, and again the sintered body was undesirable.

[0031] Comparative example 1

The water-atomised powder of SUS 316 composition used in Working example 1 was mixed and moulded as in Working example 1, except that powder with 0.35 wt% graphite powder added was used as the starting material powder. Degreasing was performed under a current of nitrogen and involved increasing the temperature from room temperature to 250°C over a period of 40 hours, increasing the temperature to

500°C over a period of 1 hour, then keeping the temperature at 500°C for 1 hour. The degreasing process was performed under a current of nitrogen, with nitrogen flowing throughout. Sintering was performed as in Working example 1, and the resulting sintered body was appraised.

[0032] Comparative example 2

A sintered body was produced according to the same processes as in Working example 1, using the water-atomised powder of SUS 316 composition with graphite added that was used in Comparative example 1, and the resulting sintered body was appraised.

[0033] Comparative example 3

A sintered body was produced according to the same processes as in Working example 1, using exactly the same compound for injection moulding as used in Working example 1, except that the degreasing was performed under a current of nitrogen with nitrogen flowing throughout; the resulting sintered body was appraised.

[0034] The appraisal results for the sintered bodies of Working example 1 and Comparative examples 1 and 2 are shown in Table 2.

[0035] In Working example 1, the degreasing process was performed according to the present invention, and so C increased after degreasing and the properties of the sintered body were desirable. In Comparative example 1, a sintered body was produced, with graphite powder added to the starting material powder for the same purpose. The properties were desirable, as with Working example 1, but the dimensional accuracy was poorer, in that the difference between the shrinkage rate in the length direction and that in the width direction was 2.5%, much greater than the 0.5% seen with Working example 1. In Comparative example 2, the

degreased body C content was excessive and a sintered body with desirable properties was not obtained. Moreover, the difference between the shrinkage rate in the length direction and that in the width direction was 3.2%, and the dimensional accuracy was poor. In Comparative example 3, the degreasing process of Working example 1 was performed under a current of nitrogen throughout and so there was little C increase during the degreasing process, the O content in the sintered body increased and a desirable sintered body was not obtained.

[0036]

[Table 1]

Table 1

	Inventive example 1	Inventive example 2	Inventive example 3	Inventive example 4 (Working example 1)	Inventive example 5	Inventive example 6
Maximum degreasing temp (°C)	550	600	750	850	1000	1050
Degreased body C content (weight%)	0.15	0.30	0.33	0.40	0.45	0.95
Sintered body C content (weight%)	0.01	0.01	0.02	0.03	0.05	0.15
Sintered body O content (weight%)	0.55	0.30	0.25	0.23	0.18	0.08

[0037]

[Table 2]

Table 2

	Sintered body density (%)	Degreased body C content (weight%)	Sintered body C content (weight%)	Sintered body O content (weight%)	Difference in length and width shrinkage rates (%)
Working example 1	97.7	0.40	0.03	0.23	0.5
Comparative example 1	97.5	0.38	0.02	0.24	2.5
Comparative example 2	95.2	0.85	0.17	0.10	3.2
Comparative example 3	96.4	0.10	0.01	0.55	0.7

[0038] Working example 2

Iron carbonyl powder and nickel carbonyl powder were used as starting materials to prepare a mixed powder of an Fe-4%Ni composition. The mixed powder had an average particle diameter of 5.5 μm , a C content of 0.05 wt% and an O content of 0.25 wt%. 9 parts by weight of organic binder of the same composition as that used in Working example 1 were added to the mixed powder, then mixing, moulding, degreasing and sintering were performed as in Working example 1, and the sintered body was appraised.

[0039] Comparative example 4

Mixing and moulding were performed as in Working example 2, except that 0.5 wt% of graphite powder was added to the Fe-4%Ni composition mixed powder used in Working example 2. Degreasing was performed under a current of nitrogen and involved increasing the temperature from room temperature to 250°C over a period of 40 hours, increasing the temperature to 500°C over a period of 1 hour, then keeping the temperature at 500°C for 1 hour. The degreasing process was performed under a current of nitrogen with nitrogen flowing throughout. The sintering was performed as in

Working example 1, and the degreased body and sintered body were appraised.

[0040] The results of the appraisals of the sintered bodies of Working example 2 and Comparative example 3 [sic] are shown in Table 3.

[0041] It was possible to obtain sintered bodies of high-density and controlled C content both by the method of the present invention and by adding graphite. However, with the added graphite method there was a large difference between the shrinkage rate in the length direction and that in the width direction, and the inventive sintered bodies had better dimensional accuracy.

[0042]

[Table 3]

Table 3

	Sintered body density (%)	Degreased body C content (weight%)	Sintered body C content (weight%)	Sintered body O content (weight%)	Difference in length and width shrinkage rates (%)
Working example 2	97.7	0.67	0.52	0.002	0.3
Comparative example 4	97.4	0.65	0.51	0.002	2.1

[0043]

[Advantages of the invention] With the production method of the present invention it is possible to appropriately regulate the post-degreasing C content and improve the dimensional accuracy of a sintered body by performing some of the metal powder injection moulding degreasing process at from 600°C to 1000°C, and by performing the heat treatment at that time in a closed heated furnace.

Translator's note

Japanese nouns can have several possible readings; common readings have been chosen throughout.

It seems likely that in section [0005], the term *houhou*, "method", is a misprint and that *houkou*, "direction", was intended.

It seems likely that in section [0040], "Comparative example 4" was intended.